Properties study of Nylon-Nanoclay composites

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Abstract: **Nanocomposite technology is a young material science and plastic nanocomposites are among its first commercial applications. Its products improve barrier, flame resistance, thermal and structural properties of many plastics. They are not only used to improve existing products, but also are extending their reach into areas formerly dominated by metal, glass and wood. Its main focuses may be the appliance, construction, electrical, food packaging and transportation sectors.**

The key to the performance of polymer nanocomposites is the quality of dispersion of the nano-reinforcement particles and the interfacial interaction between the matrix and the reinforcement phase. Optimisation of these properties requires various special and complicated treatments, which are not always easy to determine. Often property enhancement is not as significant as expected by the industry for the use of these nanocomposites.

In the study Nylon, was compounded with three different nanoclays developed and provided by CSMCRI with proportion of 3 and 5 % with and without using compatibilisers.

This study was jointly carried out by Plastic Engineering Department – Nirma University and CSCMRI, Bhavanagar. CSMCRI intended to build data and knowledge base leading to the development of the polymer / clay based nanocomposites using bentonite clay of Indian origin and had identified IDS, NU for conducting compounding and testing work in the area of "Compounding and Testing of Nanoclays for Polymer / clay Nanocomposites"

Keywords**: Nanoclay, Nanocomposite, Nylon, Polymer.**

I. INTRODUCTION:

Nanocomposite technology is a young material science and plastic nanocomposites are among its first commercial applications. Its products improve barrier, flame resistance, thermal and structural properties of many plastics. They are not only used to improve existing products, but also are extending their reach into areas formerly dominated by metal, glass and wood. Its main focuses may be the appliance, construction, electrical, food packaging and transportation sectors, but unique uses come up almost daily.

The key to the performance of polymer nanocomposites is the quality of dispersion of the nano-reinforcement particles and the interfacial interaction between the matrix and the reinforcement phase. Optimisation of these properties requires various special and complicated treatments, which are not always easy to determine. Often property enhancement is not as significant as expected by the industry for the use of these nanocomposites. The present project aims to provide an overview of the current status of these materials in terms of their development and production. In the study Nylon, was compounded with three different nanoclays developed and provided by CSMCRI with proportion of 3 and 5 % with and without using compatibilisers.

Since most polymers are hydrophobic and are not compatible with hydrophilic clays, it is often very difficult to intercalate/exfoliate clay into the polymer matrix. In such cases, pre-treatment of either the clays or the polymers is necessary.

Compatibilisers act through a chemical reaction (Reactive Compatibilisation) or through intermolecular forces of attraction such as Van der Waals, hydrogen bonding, based on polarity of the materials (Non Reactive Compatibilisation). In addition, a compatibiliser may function by more or less the same mechanism as a surfactant does to stabilize oil/ water mixture, i.e., by being soluble in one or both major components of the blend. One such mechanism is by attaching itself to one of the blend components through chemical grafting and leaving a polymeric "tail" that is soluble in the other component Stabilized, more uniformly dispersed domains result because of reduced interfacial energy between phases.

This work in the nanocomposite area is mostly confined to the laboratory level, where their structure and properties are evaluated at a fundamental level, new compositions can be developed & checked and newer applications can be explored.

II. EXPERIMENTAL

1) *Materials*

In this study the nano composites were prepared by using three nano fillers with Nylon, in proportion of 3 % and 5 %. Nylon (Gujlon) was supplied from Gujarat State Fertiliser Corporation, Baroda. Processing additives Finawax SS and G 748 were purchased form Fine organics, Mumbai for better processability of above polymers. Processing aid G 748 was added in PET and PC with proportions of 0.3 % and Finawax SS were added in nylon with proportion of 0.3 %.

Table – 2.1 General properties of polymers (Ref: From company product datasheet)

Properties	**Units**	**Standard**	**Nylon**
Tensile Strength at break	N/cm^2	ASTM D - 638	750
Elongation at break	%	ASTM D - 638	40
Flexural Strength	N/cm^2	ASTM D - 790	900
Izod Impact Strength	N cm/cm	ASTM D - 256	3.25

Optim E 117 and Optim GE 344 grafted comptibilizer purchased from Pluss polymers Ltd., Delhi. Optim E 117 was used in Nylon and Optim GE 344 was used in PC and PET. The characteristics of the comptibilizer is given in Table 2.2

Table – 2.2 Characteristics of comptibilizer used for nanocomposites

Properties	**Units**	**Values of Comptibilizer**	
		Optim E 117	**Optim GE 344**
Density	Gm/ml	0.923	0.87
MFI (190 ° 2.16 Kg)	Gm/10 min	1.5	5.5
MAH content	%	0.5-0.8	-
Grafted GMA content	%	-	2.0

The nano talc materials were used for the nano composite. These nano fillers were supplied from the CSCMRI, Bhavanagar. Nano clay 1 made by CSMCRI – Yellow in colour , Nano Clay 2, commercially available – off white in colour and Nano Clay 3, made by CSMCRI - Yellow in colour. No treatment was given to nanoclay after preparation. The general properties of nano fillers are given in Table 2.3. The particle size was measured by Laser diffraction method on Malvern's Mastersizer 2000 and surface area was measured by BET method using Nitrogen adsorption/ desorption method

Table – 2.3 the general properties of above nano fillers

Properties	**Value**
Oil Absorption:	100 to 120 % wt/wt.
Particle size:Average particle size	~15 microns and Maximum size is 35 microns.
Bulk density	0..45 to 0.50 cc/g.
Surface area:	~ 5 meter square per gram.

2) Compounding

The above polymer and nano filler were compounded in co rotation twin screw extruder (Steer Engineering make, Model: Omega 30). The granule were quenched in water and granulated. The material is dried in vacuum before injection moulding.

All compounding ingredients, such as resin, nanoclay, compatibilizer, and stabilizer can be added into the extruder main feeder. If large amount of filler, For instance, 50% of the fillers needs to be added to the formulation, split feeding of the filler will be required. It can be fed through the main feed, and the rest can be fed from the side feeder.

Table 2.4: The batch detail of Nano-composites with and without comptibilizer, (Nylon 12: 1 represents type of filler and 2 represents batch no.)

Batch code		**Filler**	**% of filler**	**Filler (Gms)**	**Polymer (Gms)**
W/o compatibiliser	**With compatibiliser**				
Nylon 11	CNylon 11	Nano clay 1	3	90	2910
Nylon 12	CNylon 12	Nano clay 1	5	150	2850
Nylon 21	CNylon 21	Nano clay 2	3	90	2910
Nylon 22	CNylon 22	Nano clay 2	5	150	2850
Nylon 31	CNylon 31	Nanoclay 3	3	90	2910
Nylon 32	CNylon 32	Nanoclay 3	5	150	2850

Table 2.5.: The batch detail of Nano-composites with using comptibilizer

Batch code	**Filler**	**% of filler**	**Comptibilizer** (gms)	**Filler (Gms)**	**Polymer (Gms)**
CNylon 11	Nano filler 1	3	90	90	2820
CNylon 12	Nano filler 1	5	90	150	2760
CNylon 21	Nano Filler 2	3	90	90	2820
CNylon 22	Nano Filler 2	5	90	150	2760
CNylon 31	Nano Filler 3	3	90	90	2820
CNylon 32	Nano Filler 3	5	90	150	2760

E.g. Nylon 12: 1 represents type of filler and 2 represents batch no.

Table 2.6: The temperature profile for compounding of composite

Screw RPM	450
Barrel zone 1	116°C
Barrel zone 2	245°C
Barrel zone 3	269°C
Barrel zone 4	269°C
Barrel zone 5	270°C
Barrel zone 6	245°C
Barrel zone 7	245°C
Barrel zone 8	250°C
Die	264°C

3) Test Sample Preparation

The granules were injection moulded on an injection moulding machine L & T (PFY40LNC4P). The mould temperature was normal in nylon.

Compounded nanocomposite granules should be added at the typical temperature setting by using recommended temperature setting of base material. The temperature range should be such to prevent thermal degradation of the products. Moderate back pressure is recommended. In addition, it should be processed with low moisture content.
The injection moulded samples as per ASTM tested for mechanical properties.

4) Testing
2.4.1 Tensile and Flexural Properties: Tensile properties and flexural properties were measured at room temperature according to ASTM standard ASTM D 638 test method using dumb- bell shaped test specimens using a universal testing machine (Lloyds). The cross head speed was 50 mm/ min, while gauge length was 65 mm.
2.4.2 Izod Impact Strength: The notched Izod impact property was measured on impact machine (Ceast make) according to ASTM D 256 test procedure. Atleast five samples were tested at each blend composition and the average value is reported. The tests were performed at room 25^0 C.

III. RESULTS AND DISCUSSIONS

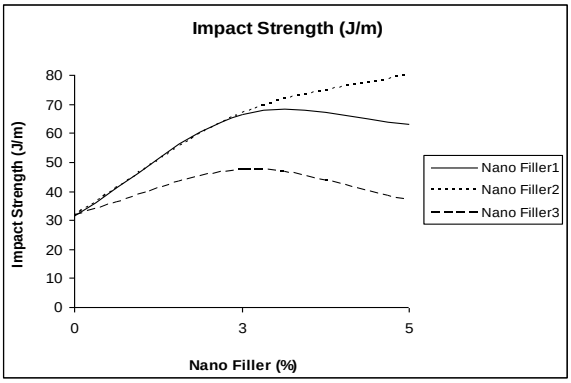
The mechanical properties of nylon nanocomposites with comptibilizer and without comptibilizer are as given in Table 3.1.

Table 3.1 Mechanical properties of nylon composite with and without using comptibilizer

Nylon nano composite without comptibilizer				**Nylon nanocomposites with compatibiliser**			
Impact Strength (J/m)							
Filler in %	**Nano Filler1**	**Nano Filler 2**	**Nano Filler 3**	**Filler in %**	**Nano Filler1**	**Nano Filler2**	**Nano Filler3**
0	31.67	31.67	31.67	0	31.67	31.67	31.67
3	66.63	67.00	47.36	3	167.19	92.61	78.91
5	62.93	79.84	36.68	5	154.18	159.84	88.59
Tensile Strength at break (N/mm^2)							
0	73.55	73.55	73.55	0	73.55	73.55	73.55
3	67.543	0	68.715	3	41.515	37.577	42.420
5	71.515	66.805	65.75	5	39.041	35.094	42.627
Tensile Elongation at Break (%)							
0	40	40	40	0	40	40	40
3	7.782	80.000	7.462	3	75.710	29.397	28.008
5	7.769	7.946	7.797	5	96.946	58.790	22.439
Flexural Strength (N/mm^2)							
0	88.26	88.26	88.26	0	88.26	88.26	88.26
3	119.41	76.01	99.24	3	45.76	66.56	55.31
5	113.00	110.39	85.91	5	47.83	57.62	54.33

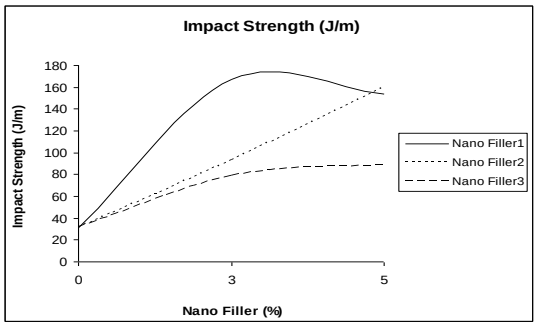
3.1.1 Izod Impact Strength

Impact strength was measured from the injection moulded article.



Graph 3.1 Impact strength of nylon nano composite without comptibilizer.

It was observed that the impact strength was improved significantly in nylon nanocomposites made from nano filler 1 and nano filler 2. But not remarkable change observed in nanocomposites made from nano filler 3.

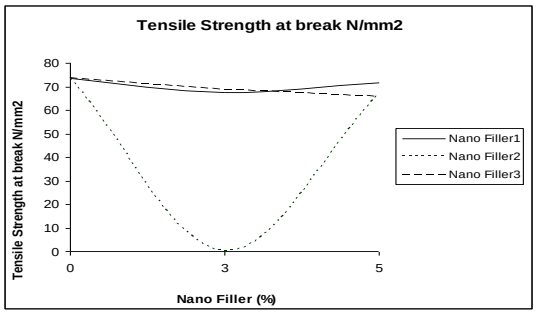


Graph 3.2 Impact strength of nylon nano composite with comptibilizer.

Graph 3.2 shows the Izod impact strength of the nano composite with comptibilizer. Impact strength increases with the addition of nano filler. The impact strength increases in nano composite made form nano filler 2 and nano filler 3 with addition. In the case of nano composite made from nano filler 1 the impact strength increases upto addition of 3 % and then decreases.

1.2) Tensile properties
The tensile properties were measured on universal testing machine by injection moulded samples as per ASTM D 256.

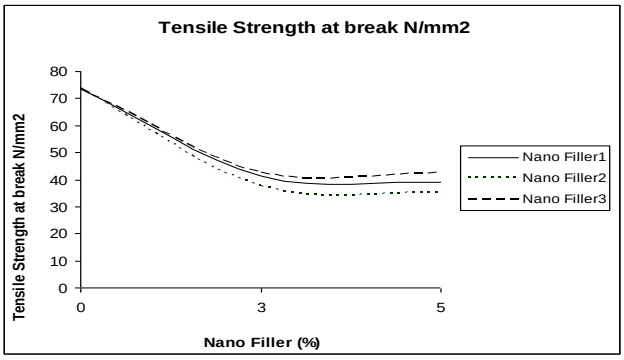
1.2.1) Tensile Strength



Graph 3.3 Tensile strength (N/mm^2) of nylon nano composite without comptibilizer.

The tensile strength of nylon nanocomposites without comptibilizer was shown in graph 3.3. The sample of nanocomposites with 3% nano filler 2 was not failed. So it

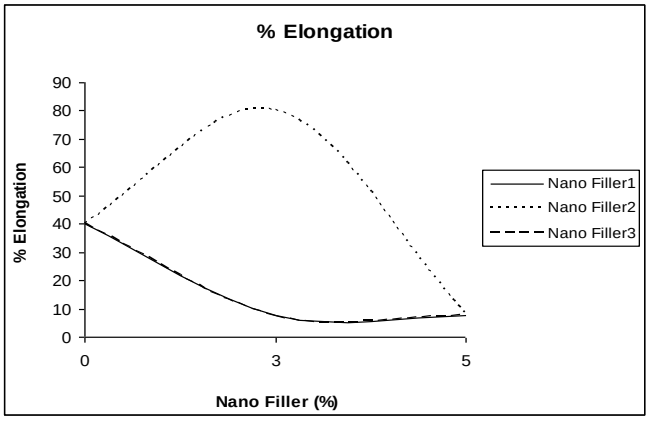
was not indicated in the result. Any significant change was not observed in the tensile strength of nano composite upto addition of 5 % nano filler.



Graph 3.4 Tensile strength (N/mm^2) of nylon nano composite with comptibilizer.

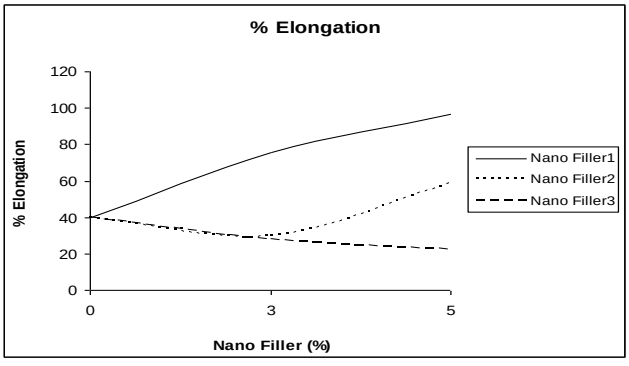
The tensile strength of the all nano composite with all type of filler decreases with addition of the nano filler. The resulted value of nanocomposites of three filler was nearby similar.

1.2.2) Tensile Elongation at Break



Graph 3.5: % Elongation of nylon nano composite without comptibilizer.

The data of nano composite with 3 %, nano filler 2 was not considered due to cold flow. The % elongation at break was decreased drastically with addition of the nano filler in all types. The results are showing the brittleness is increases with addition of the nano filler. It may be due to degradation of the treated material on nano filler.



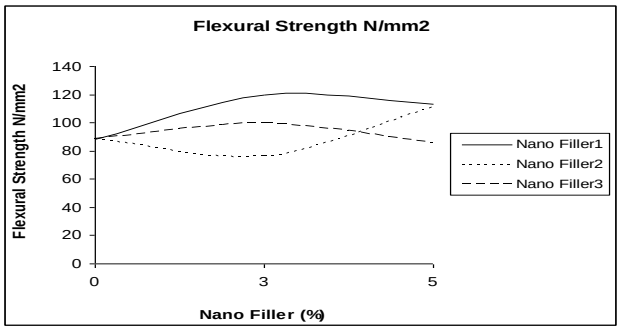
Graph 3.6 % Elongation of nylon nano composite with comptibilizer.

With addition of the compatibiliser the % elongation at break increases with addition of nano filler 1 upto 5 % addition and the % elongation decreases with addition of Nanoclay 2 and Nanoclay 3. These reduction was observed less compared to the without comptibilizer.

1.3) Flexural Properties

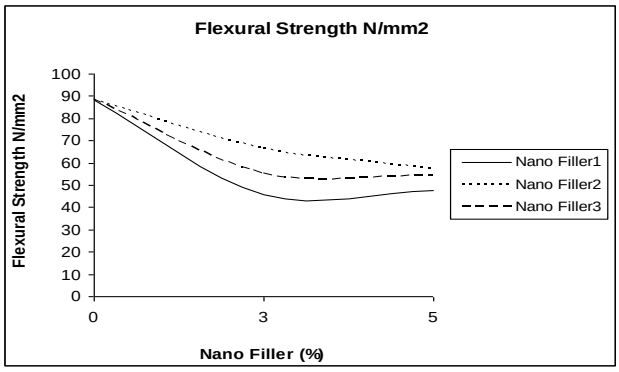
The flexural properties were measured as per ASTM standard on universal testing machine.

1.3.1) Flexural Strength



Graph 3.7 Flexural Strength N/mm^2 of nylon nano composite without comptibilizer.

The flexural strength of nanocomposites increases with addition of the nano filler 1 and nano filler 3, while the flexural strength of nanocomposites with Nanoclay 2 was increased between 3 % to 5 % addition.



Graph 3.8 Flexural Strength N/mm^2 of nylon nano composite with comptibilizer.

The flexural strength of the nanocomposites with comptibilizer is decreases with the addition of the all types of nano filler. The reduction was observed upto 3 % addition of nano filler.

IV. Conclusion

Remarks on effect of Nano clays on Nylon without using compatibiliser.

Impact Strength: it increases impact strength for nano clay 2 on increasing % of fillers. It is also recommended for Nanoclay 1 at 3% and 5%

Tensile strength at break: Not recommended with any filler.

% Elongation: Nylon not recommended

Flexural Strength: Nanoclay 1 is recommended with Nylon at 3% and 5% filler content for good Flexural strength.

Recommendations for Nanoclays without using compatibiliser

1. Nanoclay 1 is recommended with Nylon at 3% and 5% filler content for good impact strength.
2. Nanoclay 1 is recommended with Nylon at 3% and 5% filler content for good Flexural strength.

Remarks on effect of Nano clays on various polymers by using compatibiliser

Impact Strength: By adding compatibilisers it is observed that impact strength property is increased for Nanoclay 1 and Nanoclay 2.

Tensile strength @ break: With nylon not recommended with all fillers.

% Elongation: Nylon – recommended for Nanoclay 1, and continuous increase in property is observed with increasing the filler percentage in Nanoclay 1.

Flexural Strength: Nylon - Not recommended.

Recommendations for Nanoclays by using compatibiliser.

1. Nanoclay 1 is recommended at 3% and 5% filler content for good impact strength.
2. Nanoclay 1 is recommended at 3% and 5% filler content for good % Elongation.

Observations for Nanocomposites

1. All samples are opaque
2. Yellowish colour for all samples is observed.
3. CSMCRI made Nanoclay 1 is when added with Nylon without using compatibiliser at 3% and 5% filler content very good impact strength was observed but when it was compounded by using compatibiliser even better results are found hence it is recommended to use Nylon by using compatibiliser with Nanoclay 1.
4. % Elongation has been increased significantly by using compatibiliser Nanoclay 1, hence it is recommended with Nylon at 3% and 5% filler content. The graph shows continuous increase; hence it also recommended to be tested for higher % of filler content.
5. Overall very good results are observed for impact strength.
6. Recommended to use with compatibilisers.
7. Overall encouraging results for nanoclay 1 in comparison to nanoclay 3.

Future scope of work

[1] Using compatibiliser Nanoclay 1 is recommended to be tested for higher % of filler content for % elongation.

[2] By reducing particle size of Nanoclay, the whole work may be repeated.

V. REFERENCES

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